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Assessment of a novel device for onsite integrative large-volume solid phase extraction of water samples to enable a comprehensive chemical and effect-based analysis

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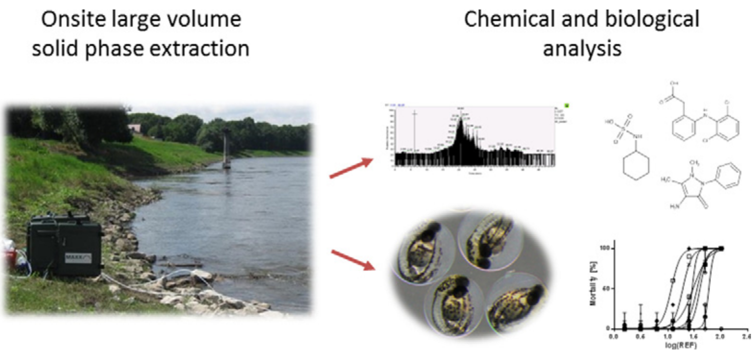
HIGHLIGHTS

- A novel solid phase extraction device for chemical and effect-based analysis was developed
- Good recoveries for organic contaminants in a large log D range were obtained for 159 out of 251 compounds
- Samples were successfully evaluated using a set of seven different bioassays for ten endpoints
- The device is applicable of sampling of up to 50 L of water

GRAPHICAL ABSTRACT



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ABSTRACT

The implementation of targeted and nontargeted chemical screening analysis in combination with in vitro and organism-level bioassays is a prerequisite for a more holistic monitoring of water quality in the future. For chemical analysis, little or no sample enrichment is often sufficient, while bioanalysis often requires larger sample volumes at a certain enrichment factor for conducting comprehensive bioassays on different endpoints or further

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LVSPE

effect-directed analysis (EDA). To avoid logistic and technical issues related to the storage and transport of large volumes of water, sampling would benefit greatly from onsite extraction. This study presents a novel onsite large volume solid phase extraction (LVSPE) device tailored to fulfill the requirements for the successful effect-based and chemical screening of water resources and complies with available international standards for automated sampling devices. Laboratory recovery experiments using 251 organic compounds in the log D range from −3.6 to 9.4 (at pH 7.0) spiked into pristine water resulted in acceptable recoveries and from 60 to 123% for 159 out of 251 substances. Within a European-wide demonstration program, the LVSPE was able to enrich compounds in concentration ranges over three orders of magnitude (1 ng L^{-1} to 2400 ng L^{-1}). It was possible to discriminate responsive samples from samples with no or only low effects in a set of six different bioassays (i.e. acetylcholinesterase and algal growth inhibition, androgenicity, estrogenicity, fish embryo toxicity, glucocorticoid activity). The LVSPE thus proved applicable for onsite extraction of sufficient amounts of water to investigate water quality thoroughly by means of chemical analysis and effect-based tools without the common limitations due to small sample volumes.

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1. Introduction

In Europe, the protection of natural water resources is regulated by the Water Framework Directive (WFD; European Union, 2000) and the Groundwater Daughter Directive to WFD (GWD; European Union, 2006) that are implemented in European member states' legislations and international river basin management. The monitoring and regulation of the chemical status of surface and ground waters refer to the priority substances listed in WFD and amended by the GWD and the Environmental Quality Standards (EQS) Directive (European Union, 2008, 2013). However, it has been demonstrated that monitoring of priority pollutants is not sufficient, because mixtures of many more known and unknown chemicals contribute to adverse environmental effects (Malaj et al., 2014; Moschet et al., 2014; Neale et al., 2015; von der Ohe et al., 2009).

The combination of targeted and nontargeted chemical screening analysis with in vitro and organism-level bioassays has been recommended for the identification of (eco-)toxicologically active compounds and mixtures by a number of more recent studies to supplement the existing concepts towards a holistic effect-based and chemical analyses approach (Altenburger et al., 2012; Brack et al., 2015; Creusot et al., 2013; Di Paolo et al., 2016; Krauss et al., 2010; Silva et al., 2002; Wernersson et al., 2015). Generally, the amount of sample enrichment required for chemical analyses and bioassay depends on the sensitivity of individual methods as well as the physicochemical properties, bioavailability, exposure concentrations, toxic potentials and mixture toxicity effects of the compounds contained in the sample. Modern chemical analytical instrumentation allows for the analysis of small water volumes with no or only low sample enrichment for most of the typical water pollutants (Bahlmann et al., 2015; Berset et al., 2010; Brack et al., 2015, 2016; Dyer et al., 2004; Fernández-Ramos et al., 2014; Seitz et al., 2006), while the analysis of some priority substances with very low EQS values as well as in vivo and in vitro tests may require greater enrichment and larger water volumes (Neale et al., 2015; OECD, 2004; OECD, 2012).

The implementation of integrated chemical and effect-based monitoring strategies (Brack et al., 2017) would greatly benefit from automated onsite sampling techniques for efficient and successful real-time collection and extraction of large water volumes. Such techniques can prevent logistic, technical, economic and scientific issues related to the storage and transport of large volumes of water to the laboratory. Furthermore, this approach allows time-integrated sampling of a water body over days or weeks to yield representative samples (Roll and Halden, 2016).

The most powerful sampling and enrichment approach for complex mixtures of known and unknown contaminants is solid phase extraction (SPE). Several well-tested and widely used solid phases that trap organic compounds with a broad range of properties (nonpolar to polar, neutral to charged) based on C_{18} or polystyrene-divinylbenzene (co-)polymers are commercially available (Fontanals et al., 2007; Fontanals et al., 2011; Hennion, 1999). A combination of complementary sorbents to cover a broad range of compounds with different properties has been successfully applied to surface water samples (Kern et al., 2009). It is an advantage of SPE to capture and stabilize the compounds on the sorbents when sampled (Hillebrand et al., 2013). Different approaches and devices for the sampling of large volumes of water have been developed since the 1970s (CIAgent, 2012; Coes et al., 2014; Dawson et al., 1976; de Lappe et al., 1983; Dean et al., 2009; Ehrhardt and Bums, 1990; Ellis et al., 2008; Gomez-Belinchon et al., 1988; Green et al., 1986; Hanke et al., 2012; Lakshmanan et al., 2010; McKenzie-Smith et al., 1994; Petrick et al., 1996; Reineke et al., 2002; Roll et al., 2016; Sarkar and Sen, 1989; SEASTAR INSTRUMENTS, 1984; Sturm et al., 1998; Suarez et al., 2006; Supowit et al., 2016; Thomas et al., 2004; Thomas et al., 2001; Weigel et al., 2001; Yunker et al., 1989). Briefly, many of the devices were best suited for low water volumes (for analytical purposes), are not (anymore) commercially available or do not operate in a fully automated mode (see Supporting material for detailed information).

Since none of the existing devices and approaches satisfies all of the above-mentioned requirements, a novel device for the onsite large-volume SPE (LVSPE) was developed. It fulfills the following technical characteristics:

- Automated device for the unattended and representative sampling according to international standards (e.g., ISO 5667-1, 2006);
- Combination of SPE with a pre-filtration cartridge to separate suspended particulate matter (SPM) from the water phase;
- Tailor-made columns that allow customizable selection and combination of sorbents to focus on chemical properties and quantities as determined by the goals of the research question;
- Implementation of a pressurized system to force the water through the extraction columns;
- Usage of 12 V electronic components (controller, pumps, valves) and low energy consumption, in such a way that the device can run with a car battery or a battery-buffered fuel cell, solar panel or wind turbine.

The successful implementation and application of sampling approaches in the chemical and biological assessment of complex environmental mixtures requires the assurance of the representativeness and integrity of the samples with minimized alteration and bias (Brack et al., 2016; Schulze et al., 2011). The aim was to assess whether the LVSPE device:

1. Is able to capture a wide-ranging set of known organic water contaminants (among them pesticides, biocides, pharmaceuticals, and artificial sweeteners) with good recoveries and high repeatability?
2. Can enrich a sufficient volume of water to perform a set of different bioassays even for minimally contaminated waters?

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